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Synthesis and characterization of highly luminescent EuOF nanoceramics by using prepared $Eu(FOD)_3$ nanoparticles via sublimation method as precursor

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Abstract

Highly luminescence europium oxyfluoride (EuOF) nanoceramics with great surface were synthesized by thermal decomposition of $Eu(OCC(CH_3)_3CHCOC_3F_7)_3$ ($Eu(FOD)_3$) nanostructures as precursor at 400 °C and ambient atmosphere without adding external additive. $Eu(FOD)_3$ nanostructures were prepared by a sublimation method at various temperatures and the effect of sublimation temperature on morphology and size of products was investigated. The as-prepared $Eu(FOD)_3$ nanostructures were characterized by a field emission scanning electron microscope (FESEM), Fourier transform infrared (FT-IR), energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction pattern (XRD). The minimum of sublimation temperature was 170 °C and the product obtained in this condition was chosen as a desired precursor to synthesize of EuOF nanoparticles. The as-prepared EuOF nano-sized ceramic was characterized by using FESEM, FT-IR, EDS, XRD and a transmittance electron microscope (TEM). The optical emission of EuOF nanoceramics was characterized by photoluminescence spectrum (PL).
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1. Introduction

In recent years, nanomaterials have been broadly investigated due to the applications in the fundamental scientific and technological interests in accessing new classes of functional materials with unique properties and applications in various fields. Also, nanostructure materials have different properties with bulk materials because their properties depends on their morphology and size [1–4].

One of the attractive rare-earth compounds is lanthanum oxyfluoride (LaOF) that they have unique properties such as excellent electric, magnetic, and optical [5,6]. Among the various LaOF, a specific attention is devoted to EuOF, due to its excellent luminescent property that using for optoelectronic devices [7], phosphors and fiber amplifier technologies [8,9], bio-imaging, bio-sensing [10,11], solar cells [12], therefore, these compounds are so attractive for scientists.

The various methods have been developed for the synthesis of lanthanium oxyfluorides (LnOF) such as: sol–gel [13], chemical vapor deposition (CVD) [14], co-decomposing the lanthanide trifluoroacetate precursors ($Ln(CF3COO)_3$) in oleic acid/oleylamine [15], precipitation [16], and solid-state reaction [17].

The preparation of nanostructure materials through solid-state thermal decomposition of organic compounds opens a new view for chemists [18–20] since there are many advantages such as: control of particle size and purity. Recently, the sublimation method was used for preparing of oxidic-compounds precursors synthesized by a thermal decomposition procedure [21,22].

In this work, for the first time, $Eu(FOD)_3$ nanostructures were prepared by the sublimation method and were used as precursor to synthesize of nano-sized EuOF ceramic through the thermal decomposition.

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2. Experimental

2.1. Materials and method

In this work, bulk $Eu(FOD)_3$ powder (100 mesh, 99.998%) was purchased from Sigma-Aldrich and used as a starting material. FT-IR spectrum was recorded on a Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range of 400–4000 cm^{-1}. Powder X-ray diffraction (XRD) patterns were collected from a diffractometer of the Philips Company with X'PertPro monochromatized Cu Kα radiation (λ=1.54 Å). Microscopic morphology of the products was studied by FESEM (Mira3 tescan) and TEM (HT-7700). The energy dispersive spectrometry (EDS) analysis was studied by a XL30, Philips microscope. Room temperature photoluminescence spectrum (PL) was recorded on a LS-55, Perkin Elmer fluorescence spectrophotometer.

2.2. Synthetic procedures

2.2.1. Preparation of $Eu(FOD)_3$ nanostructures

Preparation of $Eu(FOD)_3$ nanostructures from its bulk was performed with a cold finger set under vacuum condition. The synthetic pathway is shown in Fig. 1. Each experiment was begun with loading 0.02 g of bulk powder, which would be transferred in the bottom of external pipe. Then, the system was vacuumed. Reaction constituent was heated to reach desired temperature. The material flow is from the hot section (where the initial material is placed), to the cold area that is connected to a vacuum pump. The yellow-small depositions were collected and characterized by FESEM, FT-IR, EDS and XRD pattern.

2.2.2. Preparation of EuOF nanostructures

Nano-sized EuOF ceramic was synthesized by the thermal decomposition of $Eu(FOD)_3$ nanoparticles as a precursor that prepared via the sublimation method. 0.1 g of the precursor was placed in crucible and heated at 400 °C for 120 min. The obtained product was characterized by FT-IR, TEM, XRD, EDS and FESEM. The optical emission of EuOF nanoceramics was measured by PL.

3. Results and discussion

To investigate the effect of sublimation temperature on the morphology of europium precursor, bulk $Eu(FOD)_3$ powder was sublimated at 170, 180, and 190 °C. To fabricate EuOF nanoceramic, the as-obtained $Eu(FOD)_3$ nanoparticles (sample 1–3) were calcined at 400 °C for 120 min. The synthesis conditions of samples are illustrated in Table 1.

To produce the optimized $Eu(FOD)_3$ nanostructures as precursor to synthesize EuOF nanoparticles, the effect of sublimation temperature on the morphology of products was studied.

The sublimation process as a novel vapor-phase deposition method is vigorously dependent on the temperature. Fig. 2(a–c) shows SEM images of the $Eu(FOD)_3$ nanoparticles sublimated at 170 °C (sample no. 1), 180 °C (sample no. 2), and 190 °C (sample no. 3), respectively. The particle sizes of the samples 1, 2, and 3 are between 20–30, 40–70, and 200–300 nm, respectively, that were estimated by measuring software. Changing of sublimation temperature can be effected on morphology and size of $Eu(FOD)_3$ and EuOF nanostructures. In other word, the larger structures will be formed by increasing sublimation temperature and their morphologies will be changed from ultrafine particles to disordered structures containing larger particles. According to these SEM images, it is obvious that the smallest nanoparticles were produced when the temperature of sublimation was 170 °C, so the smallest particles obtained at 170 °C and it is considered as a desired temperature for preparation of $Eu(FOD)_3$ nanostructures.

The prepared $Eu(FOD)_3$ nanostructures (sample no. 1–3) were used as precursors to prepare nano-sized EuOF ceramics. SEM images of the EuOF nanoparticles that synthesized by the thermal decomposition route of the samples no. 1–3, are shown in Fig. 2(d–f). The aggregated nanoparticles with size about 60 nm are shown in Fig. 2d that related to sample no. 4. Fig. 2e depicts the beans-like structures with diameter and length about 50 and 100 nm, respectively, that related to sample no. 5. In Fig. 2f, SEM image of sample no. 6 is shown that the heterogeneous and amorphous structures are formed. So, the sample no. 1 was considered as desired precursor and sample no. 4 was chosen as optimum sample because its homogeneity is high and size of particles is fine. Further analyses were applied for this sample.

XRD analysis, which is the most useful technique for identification of crystalline structure, was employed to

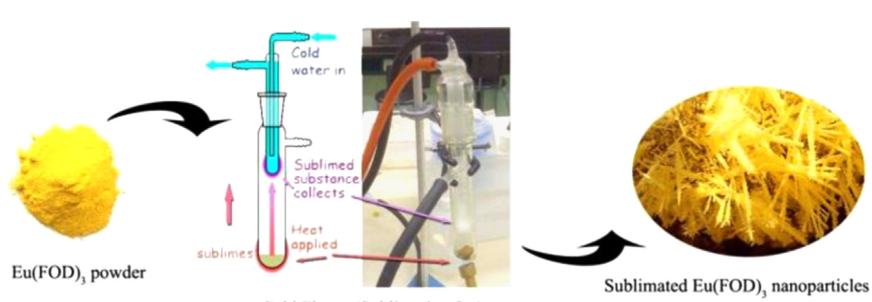

Fig. 1. The synthetic pathway of $Eu(FOD)_3$ nanostructures.

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